An Evaluation of Methods for Determining

The Linters Content of Cottonseed

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SUMMARY

The proportionate value of linters removed from cottonseed varies from about two percent to nearly twenty-five percent of the total value of products obtained from cottonseed. Although the linters content of upland cottonseed averages about 11.5 percent, it varies from 3 percent to 20 percent, representing a difference of 340 pounds per ton of cottonseed. Such variations are attributed to differences in variety and conditions of growth. These differences in linters content of individual lots of cottonseed make linters determinations an essential factor in the grading of cottonseed.

Although the present method of linters analysis was suggested by Malowan in 1920, its success was dependent upon Cox's modification in 1926, wherein hydrochloric acid was absorbed into the inside walls of a clay flower pot and the fumes forced by heat into cottonseed placed in the pot.

The objectives of this study were to provide basic information on the accuracy of available methods of analysis for linters content of cottonseed and to develop a more accurate, more rapid and more economical method of linters determination.

Principal differences in the methods of analysis are the means by which the cottonseed are fumed. Fuming can be accomplished by means of heat under pressure, under normal atmospheric conditions and in partial vacuum. In this study, fuming under normal atmospheric conditions and under vacuum were used. Under consideration were the forced-draft method, the infra-red method and the vacuum method. In the first two methods, heat is applied in a normal atmosphere by forced hot air and infra-red heat, respectively.

The vacuum method was developed in order to eliminate moisture as a factor of linters determination and to decrease errors due to seed fragmentation. Preliminary tests indicated a much better repeatability by the vacuum than by the forced-draft method.

Comparison tests made by licensed cottonseed chemists on the forced-draft and infra-red oven methods revealed a linters content standard deviation of $\pm$ 0.26 by the forced-draft method and $\pm$ 0.22 by the infra-red method on a sample of cottonseed with a moisture content of seven percent and a linters content of 12 percent. A standard error of difference of $\pm$ 0.37 was found between the two methods on nearly three thousand samples observed.

A comparison of forced-draft, vacuum and infra-red methods made by the Cotton Division, AMS, indicated that the vacuum method was considerably more accurate than the other methods. The standard deviation on all types of seed averaged $\pm$ 0.17 by the vacuum method as compared with $\pm$ 0.36 by the infra-red method and $\pm$ 0.68 by the forced-draft method.

Further work done by the Residual Lint Subcommittee, American Oil Chemists' Society, on four samples resulted in standard deviations of ± 0.18 by the infra-red method and ± 0.23 by forced-draft oven methods on low linters cottonseed. On high linters cottonseed, the standard deviations were ± 0.31 and ± 0.35 for forced-draft oven and infra-red methods, respectively. Unfortunately, no high moisture cottonseed, where the standard error is higher, were used in the comparisons. The Subcommittee did not make any tests by the vacuum method.

Although the infra-red method appeared to be superior to the forced-draft method, the vacuum method was found to be more than twice as accurate as the infra-red method.

Estimated maximum capacity of one forced-draft fuming oven and accessory equipment in an eight hour period is 105 linters determinations in duplicate, employing two operators. Two oven units may be operated by three technicians. On normal cottonseed, the maximum output on one infra-red oven unit or one vacuum oven unit in eight hours is 120 samples. Two operators are required. Three operators can handle two complete infra-red or vacuum units.

Maintenance cost of the vacuum oven and accessories is very low. Upkeep on the forced-draft equipment is also rather nominal. In the infra-red oven, however, many metal parts are exposed to acid fumes and may require considerably higher maintenance costs.

Power consumption by one unit of forced-draft equipment is estimated at about 33 kwh per eight hours at maximum capacity. Cost of gas for the fuming oven is nominal. Infra-red power consumption amounts to about three-fourths and the vacuum power consumption to approximately two-fifths of the forced-draft power requirements.

AN EVALUATION OF METHODS FOR DETERMINING THE LINTERS CONTENT OF COTTONSEED

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IMPORTANCE OF LINTERS IN COTTONSEED GRADING

For the 10-year period, 1943-53, linters have accounted for about 13 percent of the total value of products obtained from cottonseed. An annual average of nearly one and one-half million bales of linters valued at more than 50 million dollars were produced during this period. The proportionate value of linters removed from cottonseed at oil mills in the United States has varied from about two percent of the total value of products in 1920 when 70 pounds per ton were produced, to about 25 percent in 1950 when 185 pounds per ton were produced.

The linters content of upland cottonseed varies from 3 percent to 20 percent, representing a difference of 340 pounds of linters per ton of cottonseed. These variations are attributed principally to differences in varieties of cotton and environmental condition of growth. Although extreme variations are rare, differences of from two to three percent in linters content of different bale lots of cottonseed are not uncommon in contiguous areas growing a single variety of cotton. Because of these differences, it is essential that linters content be included as a factor in the grading of cottonseed.

HISTORICAL DEVELOPMENT OF METHODS FOR DETERMINATION OF LINTERS CONTENT

The use of hydrochloric acid in delinting cottonseed in the laboratory was first suggested by Malowan in 1920. 1/ This method consisted of adding dilute hydrochloric acid to cottonseed and drying on a hot plate at 105°C. Although the purpose of this method was intended to render the linters friable prior to grinding the sample for oil and ammonia analysis, Malowan pointed out that by rubbing the brittle linters off, the linters content of the seed could also be determined by weight difference.

Difficulty was experienced with the Malowan method since a portion of the cottonseed sample was charred thereby giving erratic results in the analysis of oil content of the seed. In 1926, Cox suggested a novel modification wherein hydrochloric acid was absorbed into the inside walls of an ordinary clay flower pot. 2/ When heated, the fumes were driven into the seed inside the pot, thereby chlorinating the linters.

1/ Malowan, John - Report of the Cottonseed Committee, Cotton Oil Press 4 (No. 3) March, 1920.

2/ Cox, C. H., A Method for Analysis of Cottonseed, Journal Fats & Oils Industry, III (4), April, 1926.

A laboratory method for determining the linters content on cottonseed was suggested by Rettger at about that same time. 3/ This method consisted of adding 8 to 10 drops of hydrochloric acid to a beaker and placing 10.2 grams of cottonseed on a monel metal screen over the acid in the beaker. A more rapid method of determining linters was suggested by Freyer in 1935. 4/ In this method, the linters as well as part of the seed coat were removed by concentrated sulfuric acid.

The original official USDA method of linters determination was reported by Jamieson and McKinney in 1936. 5/ The method was later revised to include a moisture determination. The authors pointed out, however, that all but about two percent of the moisture in linters was removed during the fuming. This method was approved for analyses of cottonseed under the U. S. Standards for grading cottonseed sold or offered for sale for crushing purposes. It was also adopted by the American Oil Chemists' Society and is identified as AOCS (Aa 7-44). This method, however, is time consuming, is not as accurate and is more expensive than desired.

A modification of the above mentioned method was reported by Coleman 6/, in which a 50 gram moisture sample was used rather than a ten gram sample. This 50 gram sample was placed beside the fumed sample except during the heating-fuming operation. The method proved to be more accurate than the forced-draft oven method since the large moisture sample reduced errors resulting from variation in the moisture content.

OBJECTIVES OF STUDY

The principal objectives of the present study were: (1) to provide basic information with respect to the accuracy of present laboratory methods of determining the linters content of cottonseed: (2) to develop a more accurate, more rapid and more economical method of determining the linters content of cottonseed.

APPRAISAL OF PRESENT METHODS OF ANALYSIS

Since the linters factor is included in the official U. S. Standards for analyzing and grading cottonseed sold or offered for sale for crushing purposes, an examination of all feasible method of analysis is necessary to ascertain their accuracy as a basis for determining their relative suitability for use in connection with the standards.

3/ Rettger, T. L., Journal Fats & Oils Industry, 3-135, 1926

4/ Quantitative Determination of Lint on Cottonseed, Journal Fats & Oils Industry, III (4) April, 1926

5/ Jamieson, George W. and McKinney, R. L., The Determination of Lint on Cottonseed and Cellulose in Hull Fiber - Oil & Soap XIII (6) June, 1936

6/ Private communication.

Methods for Fuming Cottonseed

Three methods for fuming cottonseed were considered: the application of hydrochloric acid fumes under pressure, under normal atmosphere and in partial vacuum. The use of hydrochloric acid fumes or hydrogen chloride under pressure presented problems, not the least of which was the danger of use of such a toxic agent under pressure. The application of the fumes under a normal atmosphere and in partial vacuum proved more successful.

The forced-draft or oil jacket oven method as developed and approved by the American Oil Chemists' Society and the USDA, relies upon heat to drive the hydrochloric acid fumes from the porous clay vessel into the mass of cottonseed, rendering the fibers friable. The infra-red oven is similar to the forced-draft oven except that infra-red heat is used instead of forced hot air.

In the vacuum method, a heat source in the base of the vacuum oven forces the hydrochloric fumes from the porous vessel into the cottonseed mass. The partial vacuum assists in liberating the fumes from the vessel and in removing the fumes and moisture from the fibers as well.

Removal of Fumed Linters From Treated Cottonseed

After cottonseed have been properly fumed, the fibers are easily removed by mechanical means or by rubbing in a cloth or plastic sheet. Although linters may be removed by hand, this method introduces a possible error due to differences in operators. Too, a mechanical method has proven to be time saving as well as assuring more uniform results.

The hand method employes a cloth or plastic sheet in which the fumed seed are placed and rubbed to remove the brittle fibers.

The mechanical method employes a brush which rubs the acid-treated seed over a standard 8" sieve (U. S. #40 or #50). There are several mechanical devices available for removing linters from cottonseed. The most commonly used method employes a bristle brush which rubs the seed over a standard #40, 8" sieve. Although this method is adequate for separating the fumed fibers from the cottonseed, there is possibility of error since it is not uncommon for one or two cottonseed or hull fragments to become lodged among the fibers of the brush. Also, a considerable amount of acidic linters dust is discharged in the air if a vacuum is not applied underneath the screens.

A novel modification of the brush method was devised by workers at the U. S. Cotton Laboratory of Stoneville, Mississippi (fig. 1). The bristle brush head was replaced by a sponge rubber disc which completely covered the sieve thereby preventing the linters dust from being discharged into the air. The center of the sieve was blocked out so that

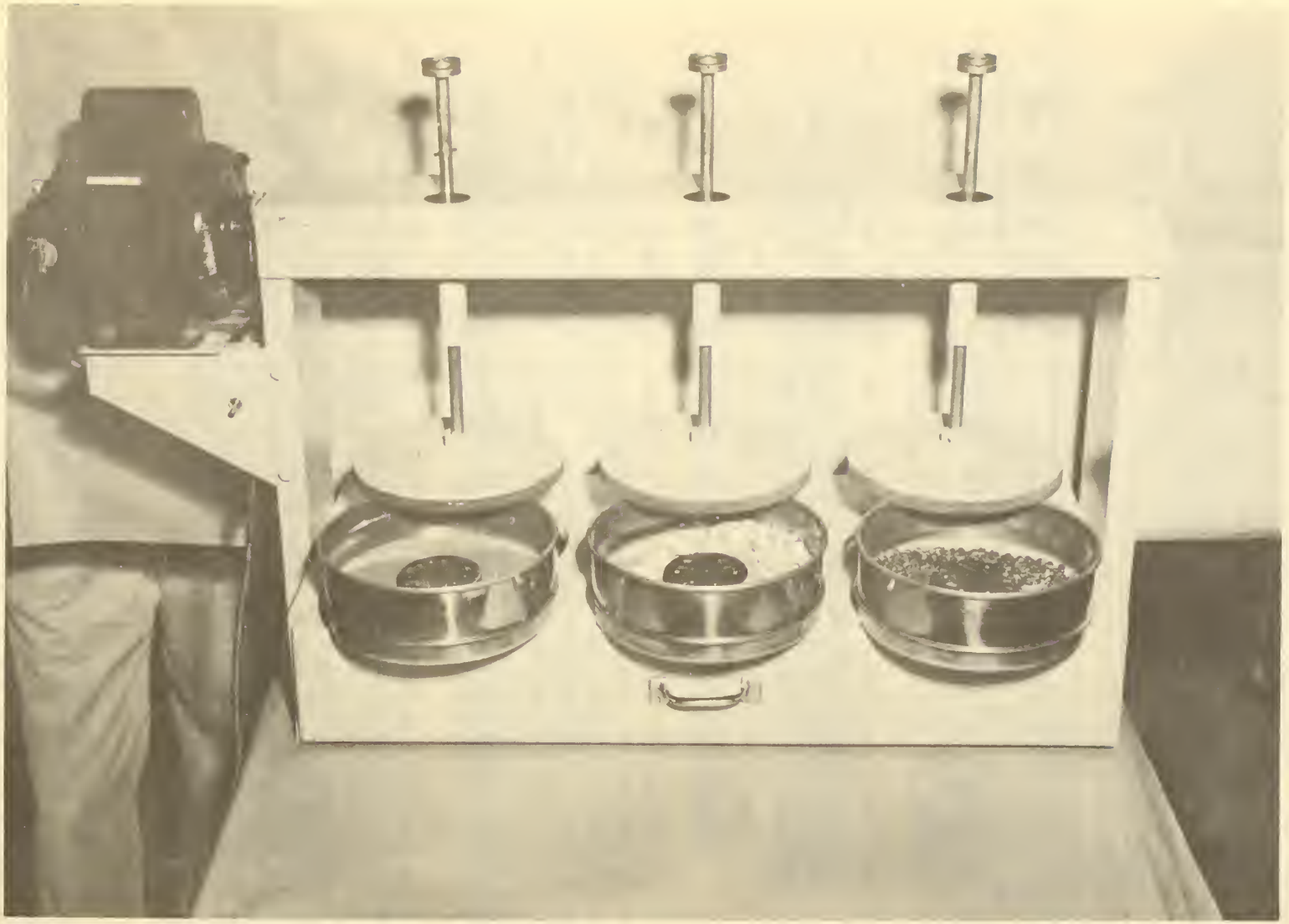

Figure 1.--Laboratory mechanical delinting device showing fumed and delinted cottonseed.

all cottonseed would be subject to approximately the same rubbing action on the outer rim of the sieve. The rubbing of the seed against screen, seed against rubber disc and seed against seed was found to completely delint the treated cottonseed in from 20 to 40 seconds. It was found that a U. S. #50 screen was better than the #40 screen for use with the sponge rubber model, especially with dry cottonseed or seed of extremely high moisture content (31-1/2 percent). The standard deviation of results using #50 screen was reduced 20 percent over that using the U. S. #40 screen.

A third method of removing fumed linters from cottonseed employs a horizontal cylinder, called the Rettger-Atkinson machine, made of 50 mesh wire screen in which two brushes rotate in a circular motion. In one model of this device, the screen cylinder is enclosed in a metal container connected to a vacuum source which removes the pulverized linters from the cylinder. This method requires about 30 seconds for delinting fumed cottonseed.

Limitations of the Forced-Draft Method

The two most serious sources of errors experienced with the forced-draft oven or AOCS (Aa 7-44) method of analysis result from improper fuming of the seed and the fracturing of the seed coats during the removal of the fumed linters. When seed are improperly fumed, no amount of rubbing or brushing can remove the fiber. If the seed coats are fractured, hull and meat fragments may be forced through the screen with the linters. (The period of time and temperature to which the samples are subjected may cause a brittleness of the seed coat).

When cottonseed are properly fumed and brushed, another serious source of error may be due to the effect of moisture on the results obtained. Since linters are more hygroscopic than the hull and kernel, the moisture content of the linters is not necessarily the same as that of the hulls and meats. 7/ The forced-draft oven method assumes that they are. Two chemists may find an equal weight of dry linters removed; however, if the chemists differ five-tenths percent in the moisture determination, there would be a difference in the linters content of the same magnitude. The Coleman method would give greater accuracy in this respect since moisture results would be more uniform; however, the 50 gram moisture samples would heavily burden the laboratories for additional oven requirements.

During the 1953-54 season, the American Oil Chemists' Society Check Cottonseed Series consisting of ten samples were analyzed for linters content for the first time. The results, using the forced-draft method,

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7/ Karon, M. L. and Adams, M. E., "Note on the Hygroscopic Equilibrium of Cottonseed Products," Journal, AOCS, XXV (1), January, 1948.

reported by chemists qualifing as licensed cottonseed chemists indicated a standard error of ± 0.36 percent. In 1954-55, the standard error was reduced to ± 0.33. This error would be reduced considerably, however, if the moisture content of the seed could be removed from consideration.

DEVELOPMENT AND EVALUATION OF VACUUM METHOD

The Fuming Vessel

Improper fuming, a common source of error, was caused in many instances by the old type vessels used in the fuming operation. These vessels have an inside diameter of about 2-5/8 inches and an inside depth of approximately 3-1/8 inches. Because of this shape, it is often difficult to obtain satisfactory fuming throughout the center of the mass of seed. Even after extended periods in the fuming oven, seed have been found to be inadequately fumed. (The quality of the clay in some vessels has been found to vary in absorptive value, requiring close observation and frequent culling to obtain satisfactory results).

If all of the cottonseed in a sample were in closer contact with the sides or base of the vessels, fuming would be faster and more thorough. Accordingly, vessels with considerably greater diameter and less depth were designed so that there were no more than about two layers of cottonseed in the vessel. A vessel about five inches in diameter and one inch in depth with a snug fitting top made of a high grade absorptive clay was designed. These vessels maintained excellent absorptiveness for twenty fumings after which they were reactivated. One series of the new type vessels were reactivated 15 or 20 times at the Stoneville Laboratory and still appeared to be as good as new.

The Vacuum Oven

Since time is an important factor, a vacuum fuming was believed to be a logical way to more quickly treat the fibers so they would become brittle and dry at a lower temperature. This lower temperature also reduces the amount of seed coat fragmentation resulting from the brushing operation.

Several vacuum ovens, using gas and electric heat sources were designed and built. Experimentation indicated that conduction of heat to the vessel directly from the oven floor was the most successful method of heat application. Small oven units which fumed six samples were used in preliminary work. Larger ovens were designed and a vacuum oven which has a capacity of 20 samples was successfully used in evaluation of the forced-draft oven, infra-red and vacuum methods. (fig. 2) A self contained aspirator unit has been developed to provide a source of vacuum (fig. 3). The twenty capacity ovens can be easily stacked, thereby conserving space in the laboratory.

Figure 2. --Technician placing flat type clay vessels containing cottonseed into the vacuum fuming oven.

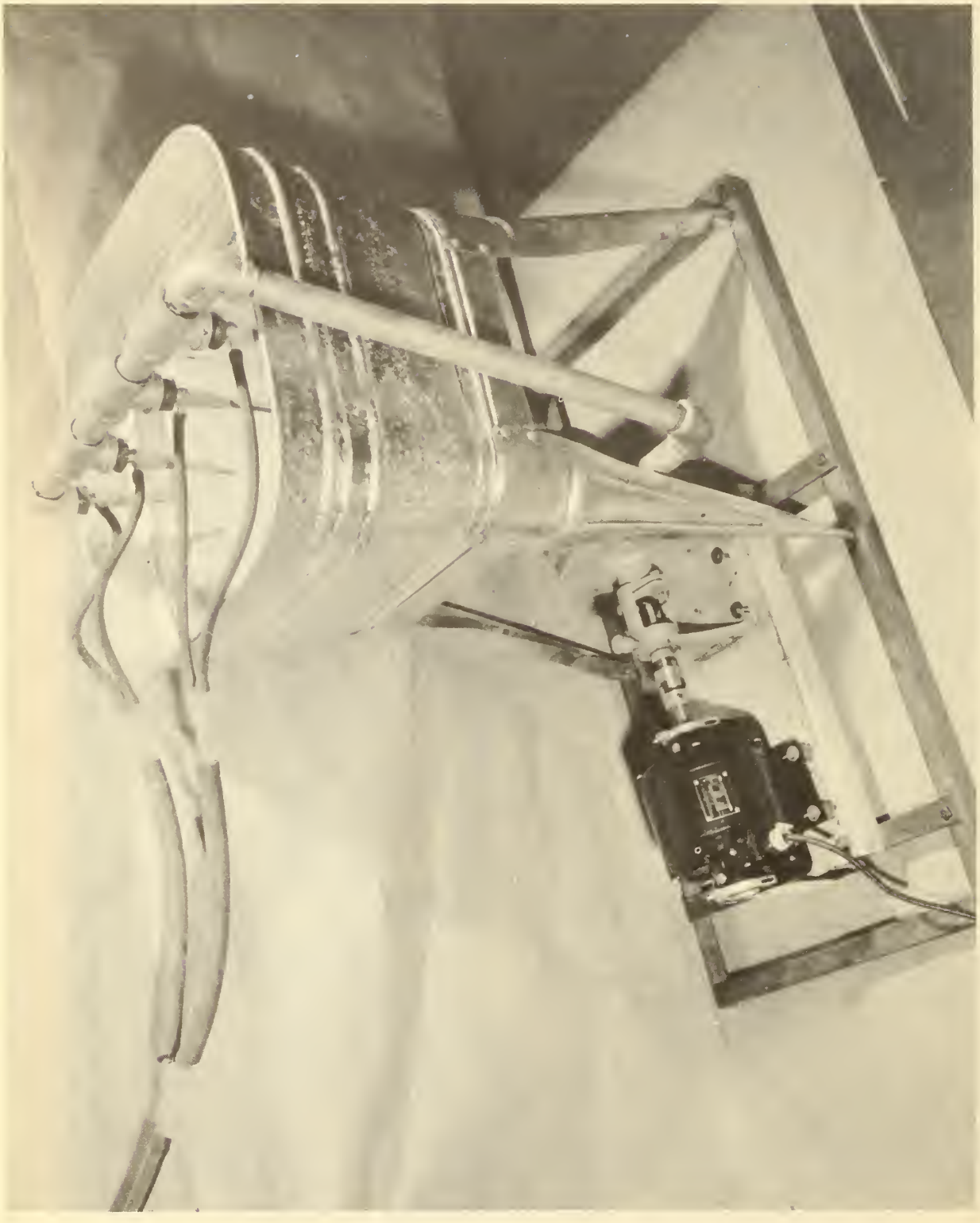

Figure 3.--This self-contained aspirator unit will provide a vacuum source for two vacuum fuming ovens.

Repeatability of Test Results and Comparisons With Forced-Draft Method

In order to determine the repeatability of results using vacuum ovens, tests were run with one oven and with two ovens. There appeared to be no difference in the accuracy when one oven or two ovens were used.

In repeatability tests, samples of cottonseed were mixed as well as possible and immediately weighed in 50 gram lots. The results of a number of these tests are found in table 1.

Table 1.--Total linters content of selected samples of cottonseed, number of replications on each lot, average moisture content and standard deviation of linters content

Sample Identification	Replications	Average Moisture Content	Average Total Linters Content	Standard Deviation of Linters Content
	Number	Percent	Percent	Standard Deviation
A	72	8.2	9.3	± 0.13
B	36	17.0	10.3	± 0.14
C	36	17.0	10.6	± 0.17
D	36	10.8	13.6	± 0.10
E	36	10.8	13.7	± 0.16

Repeatability tests were also made using the forced-draft method (AOCS Aa 7-44) and revealed standard deviations of ± 0.46 and ± 0.26 in two tests, (table 2). These observations were made on seed with slightly below normal linters content and above normal moisture content.

Table 2.--Total linters content, standard deviation, and moisture content of cottonseed analyzed by the AOCS Aa 7-44 method

Number of Replications of Sample	Average Moisture Content	Average Total Linters Content	Standard Deviation of Linters Content
	Percent	Percent	Standard Deviation
20	17.2	10.7	± 0.46
20	14.2	9.8	± 0.26

For direct comparison of the two methods, 36 replications of a sample of cottonseed were analyzed for total linters content by both the forced-draft method (AOCS Aa 7-44) and the vacuum method. The standard deviation by the vacuum method was 40 percent less than that obtained by the forced-draft method (table 3). The linters content found by that method was 0.1 percent higher than by the vacuum method.

Table 3.--Total linters content, standard deviation and moisture content of cottonseed analyzed by forced-draft (AOCS Aa 7-44) and the vacuum methods

Method	Number of Replications of Sample	Average Moisture Content	Average Linters Content	Standard Deviation of Linters Content
		Percent	Percent	Standard Deviation
AOCS (Aa 7-44)	36	17.0	10.5	± 0.24
Vacuum Method	36	17.0	10.4	± 0.14

Comparison of Results by Vacuum Method and Forced-Draft Method - (AOCS Aa 7-44)

The accuracy of results obtained by the vacuum method can best be determined by comparison with results obtained by the forced-draft method (AOCS Aa 7-44). In the 1953-54 check cottonseed series of the American Oil Chemists' Society, the analysis of cottonseed for linters content was included for the first time. Each sample provided chemists for linters determination was adequate for five or six observations. The results obtained by the vacuum method agreed closely with the accepted average of 38 collaborators participating in the check cottonseed series (table 4). In only two of the ten samples was there disagreement from the accepted average in excess of 0.1 percent.

Table 4.--Accepted average moisture and linters content of AOCS 1953-54 check cottonseed series as determined by collaborators (Method AOCS, Aa 7-44) in comparison with results obtained by the vacuum method

Sample Number	Moisture Content	Linters Content		
		AOCS Accepted Average	Vacuum Method	Difference
	Percent	Percent	Percent	Percent
1	11.9	11.1	11.3	+ 0.2
2	11.0	13.1	13.1	.0
3	8.3	10.1	10.2	+ .1
4	8.3	10.4	10.3	- .1
5	10.2	10.7	10.6	- .1
6	8.8	10.8	10.9	+ .1
7	8.7	11.3	11.4	+ .1
8	7.1	10.5	10.5	.0
9	8.7	6.6	6.6	.0
10	7.4	16.4	16.7	+ .3

COMPARISON OF RESULTS OBTAINED BY MEANS OF THE FORCED-DRAFT AND INFRA-RED METHODS OF ANALYSIS

A number of chemists licensed by the Department of Agriculture to analyze and grade cottonseed collaborated in a study to compare the results obtained by means of forced-draft ovens (AOCS Aa 7-44) and infra-red ovens in the linters determination (figs. 4, 5). (In the infra-red method, it was assumed that the linters content was the difference between fumed and delinted seed weights minus a factor representing volatile matter remaining in the linters). Analyses were made by both methods on 2,932 samples of cottonseed in one particular study. One observation by each method was made on each sample. The standard error of difference between the two series of observations was found to be ± 0.37, (table 5). Unfortunately, the range in moisture content or linters content was rather narrow for the samples available for this series. Moisture contents ranged from 6.5 percent to 11 percent while the linters content (8 percent moisture basis) varied from about 9 percent to less than 13 percent.

Table 5.--Standard error of difference between two series of linters determinations made by forced-draft oven and infra-red oven methods of analysis

Group Number	Number of Samples	Standard Error of Difference
1	2,932	± 0.37
2	243	± 0.25
3	235	± 0.27
4	466	± 0.27

Two further series of observations were made wherein standard errors of difference of ± 0.25 and ± 0.27 were found. A fourth series was made in which no correction factor was applied to the infra-red results but the linters content was found "by difference" as in AOCS (Aa 7-44); that is, linters content equals 100 minus (dry percentage of delinted seed plus volatile matter). The standard error of difference was found to be ± 0.27, indicating that there appeared to be little variability in this limited moisture and linters content range whether linters found "by difference" or "directly".

Certain of the above mentioned chemists collaborated in a further comparison of the forced-draft method and infra-red method on a uniform cottonseed with 12 percent linters content. Moisture content of the cottonseed was about seven percent. The standard deviation was found

Figure 4.--Technician removing fumed cottonseed in clay vessels from the forced-draft oven.

Figure 5.--Technician placing twenty samples of cottonseed in infrared oven for fuming.

to be slightly lower by the infra-red method than by the forced-draft method (table 6).

Table 6.--Standard deviations of linters determinations on a sample of cottonseed as made by the forced-draft method and infra-red method

Chemist	Forced-Draft Oven Method (AOCS Aa 7-44)		Infra-red Method	
	Number of Replications	Standard Deviation	Number of Replications	Standard Deviation
A	80	± 0.26	146	± 0.23
B	--	----	26	± 0.27
C	42	± 0.27	102	± 0.20
D	24	± 0.26	36	± 0.24
Average		± 0.26		± 0.22

The standard deviation of the infra-red method varied from ± 0.20 to ± 0.27 while the forced-draft method varied from ± 0.26 to ± 0.27. The standard error of difference between the two methods appears to be ± 0.34 (square root of the sum of the squares of the standard deviations by both methods). This checks rather closely with the standard error of difference of ± 0.37 found on the 2,932 samples as shown in table 5.

COMPARISON OF RESULTS OBTAINED BY FORCED-DRAFT, VACUUM AND INFRA-RED METHODS

A series of tests was made by the Cotton Division, AMS, USDA, on the three fuming methods under consideration using well known varieties of cottonseed with low, medium, and high linters content. These varieties were Deltapine 15, Stoneville 2B, and Rowden, respectively. Moisture was artifically added for the medium and high moisture tests before the samples were mixed in a paddle type mixer. Adequate time for moisture equilibrium was allowed before the tests. The large samples were again mixed before the tests.

Thirty observations were made on each sample of cottonseed for each moisture range by each method of analysis. Ten series of tests were made.

Results were tabulated two ways. In the "direct" method, it was assumed that all moisture had been removed from the fumed linters; therefore, the loss in fumed linters was assumed to be the dry linters content of the seed. The second method consisted of determining the linters content "by difference"; that is, the linters content equals 100 minus (percent of dry delinted seed plus volatile matter or moisture content). As stated previously, the latter method is used officially with the forced-draft oven both by methods approved for use by USDA licensed cottonseed chemists and by the AOCS (Aa 7-44). In this appraisal, all results will be compared with the "by difference" forced-draft results.

In the development work on linters methods, several collaborators working with an infra-red heat source developed formulas or applied correction factors to represent the volatile matter remaining in the fumed linters. In this appraisal of the three methods, no attempt is made to apply any of these correction factors since the standard deviation will be unaffected by use or omission of such factors.

Low Moisture Cottonseed

A series of 30 replications was made on low moisture cottonseed with varying linters contents (table 7). The standard deviation using the forced-draft and infra-red ovens appeared to increase as the linters content increased. In the high linters content, the standard deviation by the vacuum method was higher than for other observations by the vacuum method. However, it was considerably lower than for other methods in the same series.

Total linters content with the "by difference" forced-draft method as well as the standard deviation by this method indicate that a considerable amount of volatile matter may have remained in the seed after fuming. The average linters content by the vacuum "direct" method indicated the least variation with standard deviations of $\pm$ 0.14, $\pm$ 0.13, $\pm$ 0.27, and $\pm$ 0.14 with an average linters content which checked within 0.1 percent of the "official" forced-draft method on each series.

The infra-red "direct" method indicated smaller standard deviations than did the "by difference" method; however, the average linters content was 0.7 percent higher than the forced-draft "by difference" method in one series with medium linters content. The infra-red "by difference" method was in closer agreement on linters content but the standard deviation was greater.

Table 7.--Moisture content, linters content with standard deviation by "direct" and "by difference" methods using the vacuum, the forced-draft and the infra-red fuming ovens on cottonseed with low moisture content

Moisture Content	"Direct" Method						"By Difference" Method					
	Vacuum		Forced-Draft		Infra-red		Vacuum		Forced-Draft		Infra-red	
	Linters Content	σ	Linters Content	σ	Linters Content	σ	Linters Content	σ	Linters Content	σ	Linters Content	σ
Percent	Percent	Pct.	Percent	Pct.	Percent	Pct.	Percent	Pct.	Percent	Pct.	Percent	Pct.
6.3	9.9	±0.14	10.3	±0.33	10.3	±0.37	9.9	±0.16	10.0	±0.21	10.3	±0.37
6.2	11.1	±0.13	11.7	±0.21	11.7	±0.24	11.2	±0.20	11.0	±0.16	11.4	±0.28
6.0	15.0	±0.27	14.5	±0.56	15.3	±0.41	14.5	±0.36	15.1	±0.34	14.5	±0.36
5.0	15.2	±0.17	16.0	±0.56	15.4	±0.31*	15.7	±0.36	15.3	±0.29	15.4	±0.26*

* 8 of 30 observations omitted because of poor fuming.

Moderate Moisture Cottonseed

A series of three tests was made on cottonseed containing from 9 percent to 13 percent moisture content (table 8). There was a marked improvement in the repeatability of results by the infra-red method. The forced-draft "official" or "by difference" results were in close agreement with the "direct" vacuum method, varying only 0.1 percent. The "direct" infra-red method varied 0.3 percent in one instance from the "by difference" or official forced-draft method.

High Moisture Cottonseed

Cottonseed containing moisture in excess of 20 percent was used in the final evaluation of the three methods. The average linters content as found by the vacuum direct and both infra-red methods agreed closely with the calculated forced-draft method; however, the standard deviations of the forced-draft and infra-red methods were much higher than that found with medium and low moisture content cottonseed (table 9). The "direct" vacuum method, on the other hand, has standard deviations of $\pm$ 0.15, $\pm$ 0.13, and $\pm$ 0.17, respectively.

An examination of data on which tables 7, 8, and 9 were based reveals that sizeable and varying amounts of moisture remained in the fumed linters when analyzed by the forced-draft "direct" method. Therefore, the "by difference" AOCS (Aa 7-44) method is preferable when comparing results with the vacuum and infra-red methods.

The repeatability of the vacuum method was found, on the average, to be much better than either the infra-red or forced-draft methods (table 10). The standard deviation by the vacuum method was less than half that found by the infra-red method and less than one-third that of the forced-draft method. (The extremely high standard deviation by the forced-draft method on high moisture seed was due to the inclusion of moisture in the linters determination. This particular shortcoming was discussed earlier in this report).

Several collaborators have recommended that the fumed linters content of cottonseed minus a pre-determined factor (depending upon the moisture content of the cottonseed) be accepted as the residual linters content. Others offered a formula in which the moisture content is an integral part. Although the infra-red ovens used were practically identical, as well as fuming vessels, the length of pre-drying and fuming have varied considerably. The use of an exhaust fan to remove acid fumes, as well as to cool the oven, also may have affected results. The use of reflecting surfaces on the insides of the ovens, in an attempt to obtain uniform temperatures inside the oven, was suggested. The use of concentrated hydrochloric acid diluted with an equal part of water in fuming was reported to have reduced shattering of the delinted seed.

Table 8.--Moisture content and linters content with standard deviations by "direct" and "by difference" methods using vacuum, forced-draft, and infra-red fuming ovens on 30 replications of cottonseed with average moisture content

Moisture Content	"Direct" Method						"By Difference" Method					
	Vacuum		Forced-Draft		Infra-red		Vacuum		Forced-Draft		Infra-red	
	Linters Content	σ	Linters Content	σ	Linters Content	σ	Linters Content	σ	Linters Content	σ	Linters Content	σ
Percent	Percent	Pct.	Percent	Pct.	Percent	Pct.	Percent	Pct.	Percent	Pct.	Percent	Pct.
9.0	9.6	±0.16	10.2	±0.22	9.6	±0.26	9.7	±0.23	9.6	±0.24	10.2	±0.34
13.0	10.4	±0.16	11.1	±0.36	10.5	±0.22	10.4	±0.33	10.5	±0.30	11.0	±0.76
13.0	14.3	±0.20	14.8	±0.31	14.5	±0.38	14.4	±0.31	14.2	±0.33	14.6	±0.42

Table 9.--Moisture content and linters content with standard deviations by "direct" and "by difference" methods using vacuum, forced-draft and infra-red ovens on cottonseed with high moisture content

Moisture Content	"Direct" Method						"By Difference" Method					
	Vacuum		Forced-Draft		Infra-red		Vacuum		Forced-Draft		Infra-red	
	Linters Content	σ	Linters Content	σ	Linters Content	σ	Linters Content	σ	Linters Content	σ	Linters Content	σ
Percent	Percent	Pct.	Percent	Pct.	Percent	Pct.	Percent	Pct.	Percent	Pct.	Percent	Pct.
21	8.9	±0.15	9.0	±0.66	9.0	±0.42	9.2	±0.77	9.1	±0.99	9.0	± 0.62
22	9.9	±0.13	10.0	±0.61	9.8	±0.64	9.5	±1.05	9.6	±0.96	9.6	± 1.25
21.5	13.7	±0.17	13.8	±0.65	13.8	±0.35	14.3	±1.10	13.8	±2.09	13.6	± 1.34

Table 10.--Standard deviation of linters content as determined by vacuum "direct", forced-draft "by difference" and infra-red "direct" method of analysis, 30 replications on selected samples of cottonseed

Type Cottonseed	Forced-Draft "By Difference"	Vacuum "Direct"	Infra-red "Direct"
	σ	σ	σ
Low H_2O, Low Linters	$\pm$0.21	$\pm$0.14	$\pm$0.37
Med. Linters	$\pm$0.20	$\pm$0.13	$\pm$0.24
High Linters	$\pm$0.34	$\pm$0.27	$\pm$0.41
High Linters	$\pm$0.29	$\pm$0.17	$\pm$0.31
Med. H_2O, Low Linters	$\pm$0.24	$\pm$0.16	$\pm$0.26
Med. Linters	$\pm$0.30	$\pm$0.16	$\pm$0.22
High Linters	$\pm$0.33	$\pm$0.20	$\pm$0.38
High H_2O, Low Linters	$\pm$0.99	$\pm$0.15	$\pm$0.42
Med. Linters	$\pm$0.96	$\pm$0.13	$\pm$0.64
High Linters	$\pm$2.09	$\pm$0.17	$\pm$0.35
Average	$\pm$0.60	$\pm$0.17	$\pm$0.36

Since the pre-drying phase of the operation is time consuming and expensive, one collaborator used glass containers for pre-drying, then placing the clay tops, in which the hydrochloric acid has been absorbed, on top of the glass containers over the cottonseed. Satisfactory fuming was reported.

As suggested by collaborators who had considerable experience with the infra-red oven, a pre-drying of ten minutes duration, fuming time of 15 minutes and additional 15 minutes with the exhaust fan in operation and without the infra-red heat was used on all determinations made by this method. Surprisingly, there appeared to be little difference in infra-red average results as compared with forced-draft and vacuum results, except on low moisture medium linters cottonseed where results were 0.7 percent high (table 11). With this one exception, the results by infra-red "direct" method did not require the use of a factor. However, since the use of a factor or formula was indicated by collaborators on other tests, there is a definite need for standardization on one method.

Table 11.--Average linters content of cottonseed as determined by the forced-draft "by difference", vacuum "direct", and infra-red "direct" methods of analysis, 30 replications on selected samples

Moisture Content	Linters Content		
	Forced-Draft "By Difference"	Vacuum "Direct"	Infra-red "Direct"
Percent	Percent	Percent	Percent
6.3	10.0	9.9	10.3
6.2	11.0	11.1	11.7
6.0	15.1	15.0	15.3
5.0	15.3	15.2	15.4
9.0	9.6	9.6	9.6
13.0	10.5	10.4	10.5
13.0	14.2	14.3	14.5
21.0	9.1	8.9	9.0
22.0	9.6	9.9	9.8
21.5	13.8	13.7	13.8

Performance of the Ovens During Testing

Fuming by the infra-red oven was found to be more difficult on high linters content cottonseed. In fact, poorer results were obtained from low moisture high linters seed than with seed of higher moisture contents. As expected, shattering of the hulls occurred more frequently and to a greater extent with low moisture cottonseed. Considerably less shattering occurred during vacuum fuming as compared with the other two methods. This, therefore, partially explains the low standard deviation by the vacuum method.

Work recently completed by the Linters Analysis Subcommittee of the American Oil Chemists' Society indicates that closer agreement was found by the "direct" infra-red method than the forced-draft "by difference" method. Four standard samples of cottonseed were distributed to four collaborators for analysis by both forced-draft and infra-red methods and to nine additional collaborators for analyses by the forced-draft method. On a sample containing medium low linters and low moisture, the four collaborators obtained results which had a standard deviation of $\pm$ 0.23 by the forced-draft "by difference" method and $\pm$ 0.18 by the infra-red "direct" method (table 12). The standard deviation for all collaborators on the forced-draft method was $\pm$ 0.24.

The results obtained by the committee indicated better repeatability by the infra-red method than is shown by this study. Unfortunately, no observations were made by the Committee and other collaborators on extremely low and extremely high moisture content seed where higher standard deviations may be expected.

PRODUCTION CAPACITY OF OVEN UNITS

Forced-Draft Unit

Although the forced-draft oven (Henry design) has a capacity of 40 samples, many chemists have indicated more satisfactory fuming of cottonseed when the oven was loaded with 30 samples. (Quite often, a longer fuming period was found necessary when the oven was fully loaded with 40 samples. Frequently several samples in fully loaded ovens were improperly fumed). The oven is 40 inches high, 36 inches wide and 18 inches deep. Legs may be added or it may be placed on a table. A pre-drying of 30 minutes to one hour (dependent upon the moisture content) plus an additional hour fuming is required by the method to complete the fuming operation. Under normal operating conditions, 30 samples of cottonseed can be processed in about one hour and five minutes (when pre-drying is done in another oven) or a total of seven charges completed in an eight hour work day. This represents a total of 105 samples in duplicate. This method requires a large pre-drying capacity as well as overnight drying for the delinted cottonseed. In addition, the determination of moisture content of the cottonseed is required. Heat

Table 12.--Moisture content, linters content and standard deviation obtained by collaborators, Residual Lint Committee, American Oil Chemists' Society, 1954-55

Sample Number	Moisture Content	Linters Content		Forced-Draft				Infra-red	
				All Collaborators		Collaborators on Both Methods			
		Forced-Draft	Infra-red	Number of Observations	σ	Number of Observations	σ	Number of Observations	σ
1	7.8	7.9	7.9	170	±0.24	17	±0.23	70	± 0.18
2	7.4	19.7	19.6	124	±0.31	17	±0.32	59	± 0.35
3	6.7	13.4	13.3	135	±0.31	17	±0.26	68	± 0.21
4	7.8	14.4	14.4	131	±0.30	16	±0.26	64	± 0.25

for the fuming oven is furnished by one gas burner. A small motor powered fan forces the hot air through the oven.

Vacuum Unit

The standard vacuum oven has a capacity of 20 samples. A drying and fuming time of 35 minutes plus two minutes for unloading and reloading the oven permits 12 complete fumings per eight hour work day or 120 cottonseed samples in duplicate. The vacuum oven is very compact, measuring 32 inches in length, 25 inches in width and 6 inches in height. An allowance of six inches is made on one side of the oven for the vacuum gauge and valve. Legs only four inches long are normally used since the ovens may be stacked three high on a table. This will allow for the processing of 360 samples in duplicate in an eight hour period in a comparatively small space in the laboratory. Vacuum is normally supplied by a self-contained water aspirator unit which reuses the water and takes up all exhausted hydrochloric fumes. On cottonseed with moisture content in excess of 22 percent, an additional ten minute pre-drying under vacuum is required. Power consumption is 4,000 watts for heat (heaters "on" 20 percent, "off" 80 percent of the time) and 500 watts power for the vacuum source.

Infra-red Unit

The infra-red oven used in this work has a capacity of 20 samples. This oven measures 30 inches in height, 25 inches in depth, and 31 inches in width. Legs 18 inches or 20 inches long are used to support the oven. The heat source consists of 20 infra-red 125 watt bulbs placed directly six inches above and 20 bulbs placed six inches below the clay vessels. Power consumption is 5,000 watts, exclusive of the exhaust fan. Predrying requires 15 minutes. Cottonseed with more than 15 percent moisture must be dried an additional 15 minutes. Removal of samples to cover with acid treated ceramic vessel tops requires five minutes; fuming 20 minutes and removal of one charge and replacement with another, two minutes. This completes one cycle and requires a total time of 42 or 57 minutes (dependent on moisture content). In 8.6 hours, the oven will handle 12 charges or 120 samples in duplicate of cottonseed with moisture content 15 percent or less. Because of its bulkness, the stacking of infra-red oven units is not recommended.

Any appreciable amount of pre-drying would reduce the production rate by either the infra-red or the vacuum methods since maximum production is predicated upon the 42 and 40 minute cycles. The additional 15 minutes would reduce the output by one-third. There will be little lost time by the vacuum method, however, since few cottonseed samples exceed 22 percent moisture.

ESTIMATED EQUIPMENT AND OPERATING COSTS

Cost and Maintenance of Equipment

A large forced-draft pre-drying oven, a Henry design forced-draft fuming oven and a six unit brush machine are necessary for processing linters determinations on 105 samples of cottonseed in duplicate in an eight hour period. Estimated cost of this equipment is about $1,500.00. Initial costs for infra-red and vacuum equipment (20 unit) are estimated at about two-thirds for each of the cost of the forced-draft equipment. However, since the infra-red and vacuum methods allow for 120 duplicate determinations in 8.6 hours and 8 hours respectively, the proportionate cost based upon the output lowers the infra-red equipment cost to 62 percent and the vacuum equipment cost to 58 percent of that of the forced-draft equipment.

The present design of the infra-red fuming oven will necessitate considerable replacement of parts as exposed components of the oven are subject to acid fume erosion and, therefore, rapid deterioration. In the forced-draft oven, the fan blade and transite partitions may require replacement. Since the vacuum oven is acid-proof porcelain lined, upkeep costs should be very low.

Power Consumption

Power required for the forced-draft method (including overnight drying of the delinted cottonseed) is estimated at about 33 kilowatt hours for 105 samples of cottonseed in duplicate. Cost of gas for the fuming oven is nominal. Infra-red power consumption for 120 samples is estimated at about three-fourths and the vacuum method about two-fifths of the forced-draft requirements.

When cottonseed contain moisture in excess of 15 percent, the forced-draft method requires an additional 30 minutes pre-drying. This will add about 14 kilowatt hours. A pre-drying of ten minutes is also required for seed with moisture in excess of 15 percent by the infra-red method. Pre-drying ten minutes is also required by the vacuum method when the moisture content exceeds 22 percent.

An average of about one-tenth of all officially analyzed cottonseed contained moisture in excess of 15 percent during the past ten years, therefore, one out of ten samples would require pre-drying by the infra-red and additional pre-drying by the forced-draft method. On the other hand, an average of one-half of one percent of all cottonseed had a moisture content in excess of 20 percent. Therefore, less than one in two hundred samples would require pre-drying by the vacuum method.

APPENDIX

Methods of Analysis for Linters Content of Cottonseed

Forced-Draft Method

(Extract from USDA Official Methods of Analysis for Cottonseed)

Procedure: Dry duplicate portions of 50 grams of cottonseed, plus or minus 0.1 grams, for 30 minutes at 130°C., plus or minus 3°C., in an approved type oven. If the seed contain excessive moisture, they should be dried for one hour. Toward the end of this drying period absorb into the inner walls and bottom of a porous earthenware pot 2.0 ml. of concentrated hydrochloric acid (use 1.0 ml. for delinted cottonseed). The acid should be distributed all over the inside of the pot, and when absorbed, the inside of the pot must appear dry, otherwise, a new pot must be substituted. Place the dried seed in the pot, cover and place in an approved type of fuming oven which has been previously opened and ventilated for at least five to ten minutes, and fume for one hour. The oven temperature should gradually rise to, but not exceed, 115°C. When properly fumed, the lint should be loose and brittle, but not scorched. Transfer the treated seed to an approved sieve or screen and carefully brush with a rotating or circular motion, using a round brush, by hand, or approved machine, until all of the lint has been removed from the seed and passed through the screen. Transfer the delinted seed to a metal box provided with close fitting covers, covers removed, and place in an approved oven and dry overnight at 101°C. Remove from the oven, cover, cool in a desiccator and weigh to the nearest 0.01 grams. Determine moisture in the original seed by the same method as specified in Section 2. Calculate and report in accordance with the following example:

Example of Calculation

A equals Weight of sample (50 grams)

B equals Weight of dry, delinted seed

C equals Moisture in original cottonseed

Residual Lint, 8% moisture basis (when 50 gram sample is used)

$$\% = \frac{2 (A - B) - C}{0.92}$$

Vacuum Method

(Extract from USDA Official Methods of Analysis for Cottonseed)

Procedure: Weigh duplicate portions of 50 grams ± 0.1 of cottonseed. Absorb 2 ml. of concentrated hydrochloric acid in the bottom of vacuum type earthenware porous pots. (The acid should be distributed over the bottom of the pot. If the bottom of the pot does not appear to be dry, another pot must be substituted). Place the weighed seed in the pot, cover and place in an approved type vacuum oven. Close oven door. The temperature of the bottom of the oven will be thermostatically maintained at 175°C ±3°. After seven minutes in the oven, a vacuum of 28 ±1" Hg will be applied to the oven for 28 minutes continued treatment.

With excess moisture cottonseed (more than 22 percent), the method is modified. The cottonseed is pre-dried under full vacuum (28" ±1 Hg) for ten minutes in vessels without covers, then the hydrochloric acid is absorbed into the ceramic vessels as described above. The samples are then fumed as outlined above.

Weigh the fumed cottonseed to the nearest .01 gram. Transfer the seed to an approved delinting device for about one minute or until all linters have been removed and passed through the screen. Weigh the delinted seed to the nearest .01 gram.

Calculate and report residual linters in accordance with the following example:

B Equals Weight of cottonseed after fuming treatment

C Equals Weight of delinted cottonseed

Residual linters, 8% moisture basis, in percent equals

$$\frac{2\,(B - C)}{0.92}$$

Note: When the pots do not absorb the acid, they may be replaced with new pots or reactivated as follows:

Boil for approximately one hour in .5 percent NaOH solution. Rinse in water. Heat in a one percent solution of HCL for 20 minutes. Rinse in water. Dry overnight at 130°C. This treatment renders the pots as good as new and in some cases the absorption is actually increased.

Infra-red Method

(Proposed for inclusion in Official and Tentative Methods of the American Oil Chemists' Society by Linters Subcommittee, AOCS)

Procedure: Weigh duplicate portions of 50 grams of cottonseed $\pm$ .01 grams into a glass dish distributed evenly and dry, uncovered, for 15 minutes in the infra-red oven at $118^{\circ} \pm 3$ C. Dishes should be placed so that heat rays shall bear directly on the samples. Samples containing in excess of 15 percent moisture should be dried an additional 15 minutes.

Toward the end of the drying period, absorb into the clay cover 2.0 ml. of a dilute solution of HCL (1.0 ml. mechanically delinted seed.) At the end of the drying period, remove samples and place the cover on the glass dish with treated side toward the seed. Heat for 20 minutes using top bank of lamps only.

Remove and cool to room temperature without desiccation. Weigh seed recording weight as "A". Remove linters by use of a mechanical brushing machine recording weight of lint free seed as "B". Determine moisture in average lint removed at intervals recording moisture as "C".

Calculation: the residual linters (linters on the original seed) is conventionally calculated to an eight percent moisture basis as follows:

Residual linters, eight percent moisture basis (50 gram sample):

$$\text{Percent Linters} = \frac{2\ (A - B)\ X\ 100\text{-}C}{92}$$

Note: Large flat clay pots (inside - 6" diameter by 1-1/4" depth) have proven as satisfactory as the glass dishes and may be used.

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